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## (54) FIBRE PRODUCTS CONTAINING WATER-ABSORBENT MATERIAL

(71) I, DAVID TORR a Citizen of the United States of America, of Post Office Box 1, Oyster Bay, New York, United States of America, do hereby declare the invention for which I pray that a Patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a new and improved process of producing water-absorbent products from sheets of wood-pulp cellulose fibers. The invention also relates to new and improved water-absorbent sheets characterized by the presence therein of extending agents for the water-absorbent material present in the fibers of the sheet.

As discussed in U.S. patent no. 3,347,236, attempts have previously been made to incorporate powdered water-absorbent material into a layer of cellulose fibers, but the particles have shown an undesirable tendency to migrate during manufacture, shipment, storage or use, and attempts to fix the discrete particles by the use of adhesives resulted in considerable loss of surface area, and water absorption function.

I have discovered a simple, economical and highly effective method for anchoring the powdered material to the cellulose fibers. The method makes it possible not only to maintain the powdered material in place in a multilayer product but also to keep it fixed in a fluffy mass of cellulose fibers which is to be used, subsequently, for any desired purpose involving absorption of liquids, such as aqueous liquids and particularly body fluids (such as urine, menstrual fluid, perspiration, blood and meat juices).

In accordance with the present invention there is provided an absorbent sheet comprising an adherent blend of (a) a fluffy mass of water-insoluble fibers, (b) particles of a gel-forming material which on contact with ten times its weight of water forms a plastic gel, and (c) a silica or silicate extending agent for said gel-forming material, wherein said particles have been humidified to improve adhesion thereof to said fibers.

The invention is particularly suitable for producing disposable baby diapers. Such a diaper may comprise a plurality of layers including a water-absorbent layer, adapted to absorb and hold the urine, and a body-contacting layer which is water-permeable and permits the urine to pass through it and be absorbed in the absorbent layer. The absorbent layer may be, for example, a single relatively thick layer such as a layer of wood pulp cellulose about 1/8 inch thick or a plurality of layers. The body-contacting layer may be of porous non-woven fibrous construction and its fiber surfaces may be hydrophobic so as to reduce spreading of aqueous liquids such as urine in its major plane; for example, rayon fibers having a hydrophobic finish may be used. The disposable diaper may have an outer barrier layer which is substantially impermeable to the passage of urine or other aqueous liquids; this layer may be, for instance, a sheet of plastic such as low density polyethylene or a sheet of plastic-coated fibrous (e.g. non-woven) material. The various layers may be bonded together in any suitable manner, as by adhesive bonding along lines parallel to, and adjacent to, their edges.

The invention may be employed in a similar manner for bed pads to protect a bed surface against wetting, catamenial pads and tampons. Table napkins, bed sheets, clothing,

packaging materials, surgical pads, towelling, sutures, bandages, and similar products may also be made of the products described herein.

In the practice of the first embodiment of the invention, the present water-absorbent products are prepared by a process including the steps of (a) converting a sheet of water-insoluble fibers into a fluffy mass in the presence of particulate gel-forming material which on contact with ten times its weight of water forms a plastic gel, and a silica or silicate extending agent for said gel-forming material and (b) humidifying the particles to improve the adhesion of the gel-forming particle to said fibers.

The conversion of the sheet can be accomplished by shredding, tearing, or otherwise separating the fibers from the sheet to form the fluffy mass.

One suitable technique for incorporating the powdered composition with the cellulose fibers is to distribute the powder over a relative dense sheet of wood-pulp cellulose fibers (such as the relatively hard and brittle sheet, e.g. about 1/8 inch thick, which is the form in which cellulose pulp is conventionally supplied), and to then pass the mixture into a device of known type which tears up or macerates the dense sheet and makes it fluffy, or to add the powder to the wood pulp fibers in the macerating device, steam being injected into the device or into the resulting fluffy mass; the fluffy mass may then be passed to a drier to reduce its moisture content.

A dense cellulose sheet may have its surface portions roughened or fluffed mechanically, after which the powder may be distributed over the roughened or fluffed surface. The resulting product may be humidified before, during or after the further maceration and fluffing of the whole sheet.

Bonding by humidification can also be effected by utilizing the normal moisture content of the fibers (native cellulose fibers may have a "moisture regain" in the neighborhood of 7%) without injecting steam, by heating the mixture of powder and cellulose fibers in a closed vessel to drive off some of the moisture which, during the heat treatment or when it condenses as the blend is cooled, acts on the surfaces of the powders to aid in bonding them to the fibers.

The amount of water absorbent gel-forming material in the final product is usually within the range of 3 to 35% based on the weight of the cellulose fibers, preferably 5 or 10% to 20 or 30%. The precise amount will depend on the particular use and the total amount of cellulose fibers used in the finished product. Thus for a diaper containing 1.2 ounces of cellulose pulp, the gel-forming material may be present in amount of 0.05, 0.1, 0.2 or 0.3 ounces, for example.

Water absorbent materials which may be employed are disclosed in my copending British patent specification No. 1286679 which also discloses techniques for humidification, etc. Generally the water absorbent material will be a natural or synthetic gum or resin. Included within this group are such polysaccharides and derivatives thereof as guar gum and water-swellable cellulose derivatives such as high molecular weight cellulose ethers (e.g. sodium carboxymethyl cellulose), as well as wholly synthetic polymers. Blends of water absorbent materials may be employed, such as blends (e.g. containing about equal proportions of the materials) of sodium carboxymethyl cellulose with water soluble hydroxyethyl cellulose or with hydroxypropyl cellulose or with gelatin (e.g. gelatin A). It is desirable that the materials be harmless and inert to the body of the user of the absorbent product, and that they be substantially stable to urine (whose pH is often slightly acid, e.g. in the neighborhood of 6). Ingredients for increasing the gelling tendency may be added; thus, aluminium acetate (e.g. in amount of 5 or 10%, based on the weight of the sodium carboxymethyl cellulose) may be included.

The use of the extending agent makes possible better blending of the water absorbent particles and the cellulose fibers and, further, better flow of the powders during processing. Additionally, greater uniformity of the resulting product is realized by the use of extenders which more evenly distribute the water-absorbent material in the fiber mass. Preferred extenders are fine silica such as Cab-O-Sil, hydrous aluminum silicates, such as magnesium aluminum silicate, e.g. attapulgus clay products and the like.

The use of extenders significantly improves the present process as well as the process described in the aforementioned copending application.

The following Examples are given to illustrate this invention further. In these Examples all pressures are atmospheric unless otherwise specified. In this application all proportions are by weight unless otherwise specified.

## Example 1

2 parts of guar gum powder ("Jaguar TA") (Trade Mark) is dry blended at room temperature with 2 parts of sodium carboxymethyl cellulose powder (high viscosity type, Hercules CMC 7H4 (Trade Mark) having a degree of substitution of 0.7 and having a viscosity, as a 1% solution, of about 2500—4500 centipoises at 25°C), 1.5 parts of powdered insoluble hydrous aluminium silicate (Engelhard ASP 200 having an average particle size of 0.44 micron, the particles being flat thin plates), and 0.5 part of alkyl naphthalene sulfonate wetting agent ("Petro AG fines"). To 1.76 parts of this blend there are added a small amount of perfume (0.02 part) and 0.025 parts of a bactericidal agent (Dow

Dimaryl 200) (trade mark), a water soluble quaternary ammonium compound, namely 1 - (3 - chloroallyl) - 3,5,7 - triaza - 1,1 - azoniaadamantane chloride, which is capable of releasing 6 mols of formaldehyde per mol of the compound. The resulting dry blend, which is a free-flowing powder, is sprinkled at a weight ratio of 1:5 onto a sheet of wood pulp celulose fiber about 1/8 inch thick and the sheet is fed into a macerator of known type (such as used in the wood pulp industry) while saturated steam is led into the mass. The steaming is concluded while the fluffy fiber mass is still substantially dry to the touch. The mass may then be subjected to a drying operation.

The resulting dry product is substantially as fluffy as the mass obtained when the fibers are similarly agitated without the particles being present, and without steaming, and the two fiber masses having substantially the same appearance to the naked eye, substantially the same feel to the touch, and are both easily torn apart with the fingers. The powder-containing resulting product is found to be highly absorptive to urine. For instance when one applies urine to the product, allowing a few minutes between applications (and pressing the wet product with a spoon to simulate the effect of the movement of the baby wearing a diaper containing the product), and one then squeezes the product tightly between the fingers, no urine can be expressed from the wet product. (In addition no urine odor is evident even after hours of standing). In contrast, when the same test is applied to the untreated cellulose pulp (of a widely used commercial disposable diaper) one is easily able to squeeze out liquid urine.

Example 2

Example 1 is repeated omitting the 2 parts of carboxymethyl celulose and using 4 parts of the guar gum instead. Excellent results are obtained.

In tests of the urine-holding capacity of the guar gum and carboxymethyl cellulose alone as compared to that of blends of the guar gum and the carboxymethyl cellulose, it is found that the mixture yields, surprisingly, a somewhat more viscous gel within a given time (e.g. 10 minutes contact while stirring).

Very good results are also obtained with lower proportions of the powdered blend, e.g. 5 or 10% based on the weight of the pulp.

The composition preferably contains a wetting agent and more preferably a sulfate or sulfonate wetting agent, having a hydrophobic portion attached to a sulfate or sulfonate group, e.g. alkylnaphthalene sulfonates (e.g. sodium isopropyl naphthalene sulfonate), sodium lauryl sulfate, dioctyl sodium sulfosuccinate, or similar compounds. Among other desirable effects, the wetting agent aids in the penetration of the urine into the product and appears to give a more rapid gel formation. The composition can also contain a bactericidal agent; it has been found that the inclusion of this material greatly inhibits the formation of the undesirable ammoniacal urine odor of the urine-saturated diaper on standing.

The fluffy masses produced in Examples 1 and 2 were fabricated in conventional manners into a diaper.

The performance of my present invention involves the use of the invention claimed in my specification No. 1286679.

WHAT I CLAIM IS:—

1. An absorbent sheet comprising an adherent blend of (a) a fluffy mass of water-insoluble fibers, (b) particles of a gel-forming material which on contact with ten times its weight of water forms a plastic gel, and (c) a silica or silicate extending agent for said gel-forming material, wherein said particles have been humidified to improve adhesion thereof to said fibers.

2. A sheet as claimed in Claim 1, wherein the fibers comprise fluffed cellulose.

3. A sheet as claimed in Claims 1 or 2, wherein the extending agent is a hydrous aluminum silicate.

4. A sheet as claimed in Claim 3, wherein the extending agent is a magnesium aluminum silicate.

5. A sheet as claimed in any one of Claims 1 to 4, wherein the gel-forming material is a polysaccharide gum, carboxymethyl cellulose or a blend thereof.

6. A sheet as claimed in any one of Claims 1 to 4, wherein the gel-forming material is guar gum.

7. An absorbent sheet substantially as specifically described herein with reference to Example 1 or Example 2.

8. A process for making water-absorbent products including the steps of (a) converting a sheet of water-insoluble fibers into a fluffy mass in the presence of particulate gel-forming material which on contact with ten times its weight of water forms a plastic gel, and a silica or silicate extending agent for said gel-forming material and (b) humidifying the particles to improve the adhesion of the gel-forming particle to said fibers.

9. A process as claimed in Claim 8, wherein the water-insoluble fibers comprise cellulose fibers.

10. A process as claimed in Claims 8 or 9, wherein the finely-divided gel-forming material is applied to the sheet prior to conversion of said sheet into a fluffy mass.

11. A process as claimed in Claims 8 or 9, wherein the finely-divided gel-forming material is applied during the conversion of the sheet into the fluffy mass of fibers.

12. A process as claimed in any one of Claims 8 to 11, wherein the humidifying is effected with steam.

13. A process as claimed in Claim 12 wherein humidifying is effected during the conversion of the fibers into a fluffy mass.

14. A process as claimed in Claim 13, wherein humidifying occurs after formation of the fluffy mass of fibers.

15. A process as claimed in any one of Claims 8 to 14 wherein the resulting product is dried to reduce the moisture content thereof.

16. A process as claimed in any one of Claims 8 to 15, wherein the resulting product is formed into layers and then compacted by applying pressure thereto to reduce the thickness thereof.

17. A process as claimed in any one of Claims 8 to 16, wherein said gel-forming material is a polysaccharide gum.

18. A process as claimed in any one of Claims 8 to 16, wherein said gel-forming material is guar gum and/or carboxymethyl cellulose.

19. A process of making a water-absorbent product substantially as specifically described herein with reference to Example 1 or Example 2.

20. An absorbent sheet as claimed in any one of Claims 1 to 7 when made by a method as claimed in any one of Claims 8 to 19.

21. Table napkins, bed sheets, clothing, packaging materials, surgical pads, towelling, sutures, bandages or diapers consisting of, or incorporating, absorbent sheet material as claimed in any one of Claims 1 to 7 or Claim 20.

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